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EVALUATION OF MANGANESE PHOSPHATE COATINGS

R. A. FARRARA

FEBRUARY 1984



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US ARMY ARMAMENT RESEARCH AND DEVELOPMENT CENTER LARGE CALIBER WEAPON SYSTEMS LABORATORY BENÉT WEAPONS LABORATORY WATERVLIET N.Y. 12189

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The corrosion and wear resistance of two different manganese phosphate coatings			
with supplementary coatings of either oil or heat cured solid [ilm lubricant			
(SFL) were compared. The basic, hea	vy manganese phos	sphate was compared to man-	
ganese phosphate converted or modifi	ed via the "Endu	rion ⁹ process. The cor-	
rosion resistance of the Endurion ph	osphate was sign:	ificantly superior to the	

basic manganese phosphate. Endurion phosphate with a supplementary coating of oil did not fail after 600 hours in the 5% salt spray chamber whereas basic

20. Abstract (cont'd)

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manganese phosphate with a supplementary coating of SFL failed after 206 hours (91 - 133 hours with supplementary coatings of oil). However, the wear resistance of Endurion phosphate with supplementary coatings was approximately identical to the basic manganese phosphate with supplementary coatings.

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STATEMENT OF THE PROBLEM

The objective of this project was to compare the corrosion and wear resistance of the basic or normal manganese phosphate coating to manganese phosphate that has been modified or converted by a chemical solution named "Endurion". Supplementary coatings of either oil or solid film lubricant are applied over both types of phosphate.

BACKGROUND

The protective coating presently applied to cannon components manufactured from steel is the basic, heavy, manganese phosphate and either oil per VV-L-800 or heat cured, solid film libricant per MIL-L-46010. This correlates with Type M, Class 1, of the phosphate specification, DOD-P-16232. Oil applied to basic manganese phosphate is a relatively low cost coating that results in respectable corrosion and wear resistance whereas solid film lubricant applied to manganese phosphate is a relatively expensive coating that results in good corrosion resistance and excellent wear resistance. The modified manganese phosphate, Endurion, which correlates with Type M, Class 4 of DOD-P-16232, was selected for comparison with the present manganese phosphate because it has the potential, as described in literature, for significant improvement in performance (corrosion and wear resistance) with an associated low or small increase in cost.

Immersing steel into a hot solution of manganese phosphate results in a crystalline product that drastically reduces the electrical conductivity of the surface. Since corrosion depends upon the flow of electrons between

the anodes and cathodes (hills and valleys) that exist on a surface, decreasing the electrical conductivity of the surface will retard the corrosion process. Although the phosphate crystals help to prevent the flow of electrons, there is a space or valley that exists between the crystals of the basic manganese phosphate coating. The bottom of the space between the crystals has a very thin layer, if it exists, of non-conductive material, hence moisture that eventually goes through the supplementary coating (oil or SFL) reaches this area that is marginally protected. The result is the appearance of rust or corrosion spots after a short period of time. The Endurion process deposits particles of tin that fill or seal the space between the phosphate crystals, thereby preventing moisture from reaching the bottom of the space, thereby preventing fast corrosion or rusting problem.

The phosphate crystals are also desirable for allowing a supplementary coating to adhere to the surface. The crevices between the phosphate crystals provide space for entrapping the supplementary coating. If the supplementary coating has lubricating properties, the load carrying capacity and wear resistance of the surface will be enhanced. Since the supplementary coating is the primary provider of lubrication for wear resistance tests, the basic manganese phosphate crystal should be comparable to the "Endurion" coating. However, the tin particles provided by the Endurion process have lubricating properties; hence, improvement in the wear resistance of the final coating system could be expected.

APPROACH TO THE PROBLEM

The approach used for determining if the Endurion coating is more desirable (performance and cost) than the basic manganese phosphate was to conduct standard tests for evaluating and comparing corrosion resistance and wear resistance of protective coatings. Corrosion and wear test specimens were coated with either basic or Endurion manganese phosphate and a supplementary coating of oil or SFL was added over all the phosphated specimens. Three different oils were selected for testing in order to determine if the oil presently specified (VV-L-800) should be replaced by either oil per MIL-L-3150 (P7) or oil per MIL-L-63460 (Break-free).

a. Corrosion Test.

The accelerated corrosion test is conducted in an environment of 5% salt spray which is controlled in accordance with ASTM B 117.

The protective coating is applied to plain carbon (AISI 1020) steel panels (3" x 6" x .030" thick) purchased to Type I of ASTM D 609 (cold rolled, matte finish). The coated panels are placed in the salt spray chamber and supported in a wooden holder at a 15 degree angle from the vertical. The criterion for failure that was selected is taken from the heat cured solid film lubricant specification (MIL-L-46010) which states that failure is reached when three (3) rust spots form per panel, each of which do not exceed one (1) millimeter in length, width, or diameter. Two panels with each coating were tested until failure and the average of the two times required to generate failure is the reported corrosion resistance.

b. Wear Resistance Test.

The wear resistance test is performed on a Falex Lubricant Tester produced by Faville LeValley Corp., Chicago, Illinois. The testing is conducted in accordance with ASTM D 2625, Procedure A (Endurance Life of Solid Film Lubricants). The protective coating is applied to a cylindrical (1/4" dia. x 1-1/4" long) alloy steel pin (AISI 3135) with a hardness of $R_{\rm B}$ 80/83 and two steel v-blocks (AISI-C-1137) with a hardness of $R_{\rm C}$ 20/24. The two v-blocks are stationary and are loaded radially against the pin which is rotated at 290 \pm 10 rpm. The load is applied in increments and held for a period of time (see Table I, titled "Falex Wear Life Test Procedure") until 1000 pounds is reached. The torque required to rotate the pin is monitored at all times. If the load of 1000 lbs is reached before failure occurs, it is maintained at 1000 pounds and the time required to create failure is monitored. Failure is indicated by a torque rise of 5 inch-pounds above the steady state value. If a failure occurs before reaching the 1000 pound load, the load (not time) reached prior to failure is reported. Four tests (4 pins and 8 v-blocks) with each coating are tested to provide an average value which is the reported wear life of the coating.

RESULTS

a. Corrosion Resistance.

The data reported in Table II is the specification requirements for the corrosion resistance of basic and Endurion manganese phosphate with and without supplementary coatings. This data is summarized to provide a

feel or basis for comparing the phosphate coatings and the supplementary coatings. This data reveals that the Endurion process is expected to provide better corrosion resistance than the basic manganese phosphate and that a supplementary coating, especially solid film lubricant, will improve corrosion resistance significantly.

The data reported in Table III (5% Salt Spray Test) is actual test data which is the average number of hours required from two (2) steel test panels with each coating to result in failure (three (3) rust spots per panel). The data clearly indicates that the supplementary coatings (oil or SFL) provide the primary resistance to corrosion. The basic manganese phosphate fails after approximately 1-1/2 hours and the Endurion manganese phosphate fails after approximately 24 hours if they are not protected with a supplementary coating. The primary result from the test is that converting manganese phosphate via the Endurion process drastically improves the corrosion resistance. Applying oil to the basic manganese phosphate resulted in failure, depending upon the type of oil, after approximately 100 hours (range of 91 for VV-L-800 and 133 for MIL-L-63460), whereas the panels coated with Endurion manganese phosphate and oil did not show any signs of rust after 600 hours of testing. Applying heat cured solid film lubricant over the basic manganese phosphate improved the corrosion resistance (206 hours) but this is significantly less than the Endurion phosphate plus oil (600+).

b. Wear Resistance.

The data reported in Table IV (Falex Test) is the maximum load reached for coatings that failed prior to the load reaching 1000 pounds or the time required to create failure at the maximum load of 1000 pounds. The data clearly reveals that the load carrying capacity of oils is significantly less than solid film lubricant, hence the wear resistance of solid film lubricant is far beyond oils. Also, converting phosphate \ the Endurion process did not significantly improve wear resistance (s' ht improvement with oils; slight degradation with solid film lubricant). .' is explained by the reasoning that the role of the phosphate is to provide numerous cavities which trap the supplementary coating of oil or SFL. The Endurion coating has tin deposited in the cavities which reduces the space for the supplementary coating. The supplementary coating is the primary vehicle for resisting wear, and if there is some space remaining in the cavities after the Endurion process, the supplementary coating will remain for resisting wear. While the tin supposedly acts as a lubricant, once the supplementary coating is removed or breaks down, the tin does not prevent failure.

c. Cost Comparison.

The yearly cost of material and labor that is typically expended at Watervliet Arsenal for applying basic manganese phosphate and solid film lubricant is reported in Table V. The estimated cost for installing and applying Endurion process is also reported in Table V. This data, furnished by the Operations Directorate, Watervliet Arsenal, reveals the following: (1) the cost of chemicals for the Endurion process would

increase the overall expense for phosphating by approximately 25., (2) the Endurion process would not require any measurable increase in labor cost, and (3) the total cost for applying solid film lubricant is more than the cost for applying the Endurion coating.

CONCLUSIONS

- a. A coating of manganese phosphate converted by Endurion and coated with oil provides excellent corrosion resistance for steel as measured by 5° salt fog test.
- b. The oil per MIL-L-63460 provided the highest corrosion resistance when applied to the basic manganese phosphate. However, all three oils tested provided over 600 hours when applied to the Endurion phosphate.
- c. Solid film lubricant can withstand high loads (1000 lbs) and will break down only after a considerable length of time (minutes) as measured by the Falex test.
 - d. Oil cannot withstand high loads as measured by the Falex test.
- e. Converting manganese phosphate by the Endurion process does not significantly improve wear resistance.
- f. Solid film lubricant applied over regular manganese phosphate provides better corrosion resistance than oil applied over regular phosphate but has significantly less corrosion resistance than oil applied over converted manganese phosphate.
- g. The cost for applying the Endurion process is not large when compared to the cost of applying the basic manganese phosphate (increase of approximately 25%) and the cost for applying solid film lubricant is somewhat more than the Endurion process.

TABLE I

Falex Wear Life Test Procedure (0-3000 in.-lb. Gage)

		Time		
		To Reach or Hold Load	Cumulative Total	
1.	Increase load from 0 to 300 lbs.	25 secs.	25 secs.	
2.	Hold at 300 lbs.	3 mins.	3 mins25 secs.	
3.	Increase load from 300 to 500 lbs.	20 secs.	3 mins45 secs.	
4.	Hold at 500 lbs.	l min.	4 mins45 secs.	
5.	Increase load from 500 to 750 lbs.	25 secs.	5 mins10 secs.	
6.	Hold at 750 lbs.	l min.	6 mins10 secs.	
7.	Increase load from 750 to 1000 lbs.	25 secs.	6 mins35 secs	
8.	Hold at 1000 lbs. until failure			

- a. Failure is indicated by a torque rise of 5 inch-1bs above the steady state torque value or breakage of the shear pin.
- b. Both pin and V-blocks are coated with SFL.

TABLE II

Specification Requirements for Corrosion Resistance (5% Salt Spray) of Manganese Phosphate with and without Supplementary (Supril) Coatings

Type of Coating	Specification	Requirement
Basic Manganese Phosphate without Suppl coating	DOD-P-16232 Type M, Class 3	1-1/2 hrs.
Basic Manganese Phosphate with oil per MIL-L-3150	DOD-P-16232 Type M, Class 2	24 hrs.
Basic Manganese Phosphate with solid film lube per MIL-L-46010	MIL-L-46010	100 hrs.
Endurion without Suppl coating	DOD-P-16232 Type M, Class 4	24 hrs.
Endurion with Suppl coating (suppl coating is not specified)	DOD-P-16232 Type M, Class 4	72 hrs.

TABLE III

Corrosion Resistance (5% Salt Spray) of Basic Manganese and Endurion Phosphate With and Without Supplementary Coatings

Supplementary Coating	Basic Manganese Phosphate (Type M, Class 1)	Endurion (Type M, Class 4)
0il per VV-L-800	91	600+
Oil per MIL-L-3150	122	600+
0il per MIL-L-63460	133	600+
Solid Film Lube per MIL-L-46010	206	744+
No Supplementary Coating	1-1/2 - 3	24 - 30

TABLE IV

Wear Resistance (Falex Test) of Oils and Solid Film Lubricant (Heat-cured)
Applied to Manganese Phosphate (Type M, Class 1 and Type M, Class 4)

Supplementary Coating	Basic Manganese Phosphate (Type M, Class 1)	Endurion (Type M, Class 4)	
	(Failure load or time for failure at 1000 pound load)	(Failure load or time for failure at 1000 pound load)	
Oil per VV-L-800	500 lbs - 4 tests	500 lbs - 2 tests	
		750 lbs - 2 tests	
Oil per MIL-L-63460	750 lbs - 2 tests	750 lbs - 1 test	
	1000 lbs/15 sec - Avg of 2 tests	1000 lbs/20 sec - Avg of 3 tests	
Solid Film Lube per MIL-L-46010	1000 lbs/138-1/2 min - Avg of 4 tests	1000 lbs/74 min-10 sec - Avg of 4 tests	

TABLE V

Cost Comparison of Basic Manganese Phosphate, Endurion Phosphate, and Solid Film Lubricant (Heat-Cured)

NOTE: The cost values are based on typical actual yearly costs at Watervliet Arsenal.

	Material	Labor	<u>Total</u>
Basic Manganese Phosphate	\$33,841	\$60,195	\$94,036
Solid Film Lubricant for Breech Mechanisms (cost of energy for curing is not included)	\$ 7,910	\$23,580	\$31,490
Endurion Phosphate	\$24,300	(5 minutes per load - accomplished by personnel operating basic phosphate process)	\$24,300

Initial Cost to install Endurion Process - \$50,000

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